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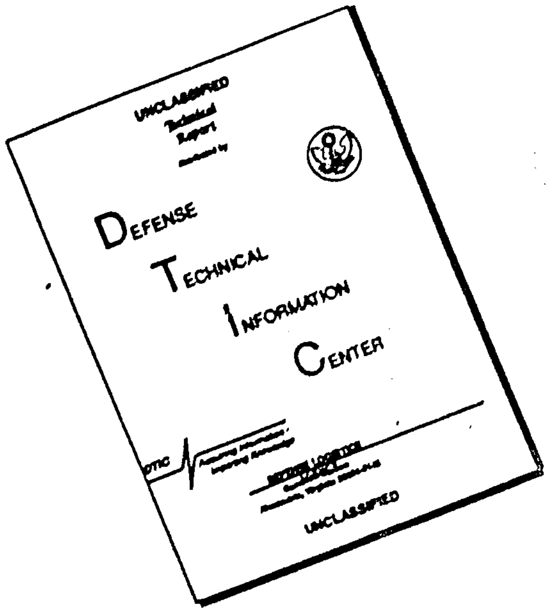

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WAL 130/24

WATERTOWN ARSENAL
LABORATORY

MEMORANDUM REPORT

NO. WAL 130/24
O.O. Project TB4-120H

METALLOGRAPHIC METHODS

Polishing Procedure for Metallographic Examination of Cemented Carbides

BY
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DATE 31 January 1949

WATERTOWN ARSENAL
WATERTOWN, MASS.

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Memorandum Report Number WAL 130/24

O.O. Project Number TB4-120H

METALLOGRAPHIC METHODS

Polishing Procedure for Metallographic
Examination of Cemented Carbides

WATERTOWN ARSENAL LABORATORY

Authorized by: Ordnance Department Order #80 31 January 1949
O.O. Project Number: TB4-12CH
Memorandum Report Number: WAL 130/24
Priority: 3B
Title of O.O. Project: Metallographic Methods
WAL Project Number: 14.10-U

TITLE

METALLOGRAPHIC METHODS

Polishing Procedure for Metallographic
Examination of Cemented Carbides

OBJECT

To establish a satisfactory procedure for polishing cemented carbide specimens for metallographic examination.

SUMMARY

Search of the literature and consultation with representatives of manufacturers of cemented tungsten carbide tool tips revealed a wide variety of polishing procedures for the preparation of metallographic specimens of cemented carbides. After careful study of the variations in these polishing techniques, a method was evolved which, it is believed, embraces the best portions of these procedures. This method is capable of producing a polished surface on cemented carbides that is free of scratches at a magnification of X1500. Porosity is revealed by photographing the unetched surface at X250 or X1000 and the microstructure by etching in alkaline potassium ferricyanide and photographing at a magnification of X1500.

CONCLUSIONS

1. The technique finally adopted utilizes the following sequence of operations:

   a. Grinding by means of a silicon carbide wheel with the specimen flooded with a copious supply of a water solution of soluble oil.

b. Manually dry polishing on two grades of boron carbide polishing papers with added boron carbide grit.

c. Finish polishing wet on diamond impregnated, horizontally rotating, cloth covered wheels. Three types of cloths are used in sequence: silk, wool broadcloth, and finally Selvyt or Gamal cloth.

2. The technique adopted permits:

a. Producing of scratch free surfaces

b. Retaining of inclusions

c. Minimizing of relief polishing

d. Revealing porosity.

e. Revealing constituent grains when samples are etched with alkaline potassium ferricyanide

f. Identifying constituent grains in some instances.

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INTRODUCTION

This Laboratory is frequently called upon to report on the microstructure of cemented carbide tool tips and HVAP tungsten carbide cores. Due to the extreme hardness of cemented tungsten carbide specimens, difficulties have been experienced, even after long preparation times, in producing good surfaces for metallographic examination.

The purpose of this investigation was to develop a procedure for producing a good quality polish (free from scratches, pits, etc.), economically and in a reasonable length of preparation time using standard polishing equipment found in all metallographic laboratories.

A review of the literature indicates that cemented carbides are polished on various types of metal or wooden laps with boron carbide and/or diamond powder of different degrees of fineness. For example, Schröter[1] employed carborundum powder followed by diamond powder on steel laps and with a finer grade of diamond powder supported on a felt pad for the final polishing. After initially grinding with a silicon carbide wheel, Hoyt[2] used boron carbide on copper laps and fine diamond powders on wooden laps for final polishing. The use of felt disks impregnated with diamond powders is mentioned by Grodzinski[3]. MacKenzie[4] and Engle[5] recommend the use of boron carbide followed by diamond powder. Shute[6] employs hard wooden laps soaked with olive or nut oil impregnated with several grades of diamond powder for final polishing. Norton[7,8] at this Arsenal initially prepared samples with a diamond wheel followed by polishing on cloth covered disks using various grades of diamond powder.

Three large American manufacturers, The Carboloy Company, Inc., Fansteel Metallurgical Corporation, and Kennametal, Inc., vary widely in their methods of preparing metallographic samples. They grind on diamond wheels and polish on laps made of bronze, lead-tin alloys, or paper and use various grades of diamond powders. Details of their methods are given in the Appendix.

To select a sequence of operations that would provide a satisfactory finish with a minimum of effect and expense, the various suggestions made in the literature, and by the above manufacturers, were tried out, using eight grades of cemented carbide tool tips for specimens. The procedure that was eventually evolved is given below in detail, but the experimental data that led to the selection of the particular sequence of operations that was finally selected are omitted.

POLISHING PROCEDURE

Samples of eight typical commercial grades of cemented carbides were obtained which contained various amounts of tungsten, titanium, columbium, tantalum, carbon cobalt, and iron. The chemical analyses, nominal composition by weight and the areas of polished surfaces are given in Table I.

The standard cutting, grinding and metallographic polishing machines were readily adapted for the preparation of the samples.

1. Initial Cutting. A diamond cut-off wheel was used for cutting the specimens to suitable size, using plenty of water soluble oil solution.

2. Mounting. All specimens were mounted in unhardened medium carbon steel clamps.

3. Surface Grinding.

   a. The specimens were ground with a freshly dressed 10" diameter "green" silicon carbide wheel (Norton Grinding Wheel #39C80 - 17V), using plenty of water soluble oil solution and a spindle speed of 3450 rpm.

   b. The specimens were then reground with a 10" diameter 100 grit diamond grinding wheel (Norton Type #D-100-J-100-B 1/16 shape DIT), using plenty of water soluble oil solution and a spindle speed of 3450 rpm. (It should be pointed out that fine uniform cuts are essential in the grinding operations.)

4. Dry Polishing

   a. The specimens were polished by hand on #320 silicon carbide or boron carbide polishing paper placed on a glass plate. A small amount of 600 grit boron carbide powder (Norton Company's "Norbide") was sprinkled on the polishing paper.

b. The specimens were then repolished by hand on #400 silicon carbide or boron carbide polishing paper placed on a glass plate. A small amount of 800 grit boron carbide powder (Norton Company's "Norbide") was sprinkled on the polishing paper.

In these operations it is essential that the polishing paper be kept clean and the abrasive grit sharp.

5. Wet Polishing. Diamond dust held by a slightly moistened cloth is very satisfactory for the final polishing steps.

Diamond powders supplied by Arthur A. Craft Company, Boston, Mass. were used and are reported to have the following particle sizes:

#120	diamond dust	- 150-170 mesh
#5	diamond dust	- 13-37 microns
#7	diamond dust	- 2 and finer microns

Tiny pinches of the diamond dust have been found sufficient for polishing. The following method for adding this tiny pinch has been found useful:- shake the corked tube containing the dry diamond powder against the stopper and return the tube to the upright position. Remove the cork stopper to which fine particles of the diamond powder have adhered, then by tapping the stopper over the center of the disk a very small quantity is transferred to the polishing cloth. Further additions of the diamond abrasive may be added in this manner to the polishing cloth as needed.

a. The samples were first polished on a silk or rayon covered 8 or 10" diameter polishing disk, moistened by a few drops of water. A tiny pinch of Craft's #120 diamond powder was sprinkled on the center of the covered polishing disk. The sample was polished near the center of the disk which was rotating at about 50-100 rpm.

b. The samples were then polished on a silk or rayon covered 8 or 10" diameter polishing disk, moistened by a few drops of water. A tiny pinch of Craft's #5 diamond powder was sprinkled on the center of the covered polishing disk. The sample was polished near the center of the disk which was rotating at about 50-100 rpm.

c. The samples were finally polished on a polishing disk covered with the finest grade of broadcloth, Beuhler "Selvyt" #1565 AB, or Fisher "Camal" cloth #12-282, moistened with a few drops of water. A tiny pinch of Craft's #7 diamond powder was sprinkled on the center of the covered polishing disk. The sample was polished near the center of the disk which was rotating at about 50-100 rpm. The samples were washed and dried in an air blast.

It is recommended that the samples be examined under the microscope at a magnification of X250 for deep scratches during the wet polishing stage using #5 diamond powder and after polishing with #7 diamond powder, the sample should be scratch-free at a magnification of X1000.

Relief polishing may result from using a polishing cloth that is too dry. If the phases appear in relief, it is recommended that the sample be surface ground once more and repolished. The structural composition of certain samples will cause some phases to show in relief even with the best polishing technique.

If the polishing cloth is too wet it will cause pitting; also, the diamond abrasive will be washed off and lost.

Photomicrographs were taken at a magnification of X250* to show the average amount of coarse porosity present in each sample, Figs. 1, 4, 7, 10, 13, 16, 19, 22, and at a magnification of X1000* to reveal the average amount of fine porosity, as well as non-metallic inclusions, Figs. 2, 5, 8, 11, 14, 17, 20, 23.

Etching of the polished surfaces presented no difficulty. All samples were etched in hot alkaline potassium ferricyanide etchant containing 10 grams of potassium hydroxide, 10 grams of potassium ferricyanide and 100 cc water. This solution is heated to simmering, NOT to vigorous boiling. Photomicrographs were taken of the phases present at a magnification of X1500*, Figs. 3, 6, 9, 12, 15, 18, 21, 24.

DISCUSSION

The polishing method described in this report embraces portions of methods found in the literature and in use by American manufacturers, with some modifications. It can readily be applied to standard polishing equipment found in metallurgical laboratories.

---

*Bausch & Lomb Research Microscope

X250	Objective X21	(.40 N.A.)	Eyepiece	X7.5 (Hyperplane)
X1000	Objective X58	(.85 N.A.)	Eyepiece	X10 (Hyperplane)
X1500	Objective X80	(1.40 N.A.)	Eyepiece	X10 (Negative)

All photomicrographs were taken with this equipment.

It was found that this procedure produces a polished surface which is free from scratches and pitting as seen under high magnification. The samples can be polished in a reasonable preparation time and are suitable for estimating coarse and fine porosity and nonmetallic inclusions, as shown in Figs. 1, 2, 4, 5, 7, 8, 10, 11, 13, 14, 16, 17, 19, 20, 22 and 23.

It is believed that the polishing method described herein is superior to polishing on various metal, wooden, or other types of laps impregnated with diamond powder, since it was observed that the latter methods frequently result in pitting and fine scratches which are revealed at magnifications of X250 and X1500.

The use of cloth covered disks, on which is sprinkled a judicious amount of diamond powder, presents no difficulty. On the other hand, it is believed the employment of metal and other special types of laps may involve some problems, namely, procurement and the care and preservation of the lapping surfaces.

The photomicrographs of the unetched samples, polished by the method described herein, reveal in a few cases slight relief polishing. This may be due to the composition of the phases present in the samples, as this relief polishing persisted after regrinding and repolishing. It appears in Figs. 5, 8, 11, 14, 17, 20 and 23.

The resulting polished surfaces of the samples investigated were satisfactory for clearly revealing the shape and size of the carbide grains and the several phases present after etching in hot alkaline potassium ferricyanide. In the photomicrographs, Figs. 3, 6, 9, 12, 15, 18, 21 and 24, a typical tungsten carbide grain is indicated by arrow "1" and a solid solution phase by arrow "2". The cobalt binder appears as a fine white area surrounding the tungsten carbide and solid solution grains.

It has been stated in the literature that difficulty has been experienced in the identification of the phases in this type of material since it has been shown that during the sintering operation all the TaC, CbC, and TiC form a single phase and also this phase will take in solid solution a considerable amount of WC[9]. This has been confirmed in the photomicrographs described in this report since the samples examined containing various amounts of Ta, Cb, and Ti show only an angular tungsten carbide phase, a rounded solid solution carbide phase and the cobalt binder.

Studies are in progress on the metallographic studies for distinguishing the various carbide phases present in cemented carbides[10].

Recently, a metallographic method has been developed for the investigation of cemented carbides, particularly of the system tungsten carbide-titanium carbide-cobalt[10]. A selective etching reagent is applied which permits the distinction of different carbide phases which might be present in this particular system.

TABLE I

CHEMICAL ANALYSES AND SIZE OF SAMPLES

Sample Number	Elements Present - Per Cent by Weight							Nominal Composition - Per Cent by Weight						Area of Polished Surface Sq. in.
	W	C	Ti	Cb	Ta	Co	Fe*	WC	TiC	CbC	TaC	Co	Fe	
1	86.65	5.58	—	--	--	6.65	1.01	92.34	--	--	—	6.65	1.0	.094
2	76.10	5.85	4.01	--	.79	11.00	2.01	80.50	5.5	—	1.0	11.0	2.0	.157
3	67.75	6.72	7.12	5.92	3.12	8.33	1.20	71.70	8.3	7.0	3.5	8.3	1.2	.063
4	63.30	5.95	1.27	11.85	3.28	12.74	1.40	67.5	1.6	13.0	3.5	12.74	1.4	.157
5	63.02	6.49	7.20	6.26	5.34	10.22	1.24	67.2	9.0	6.8	5.6	10.22	1.2	.125
6	62.24	7.24	8.88	10.60	—	9.36	1.46	66.4	10.3	12.5	—	9.36	1.4	.125
7	59.89	6.72	6.40	10.38	—	15.25	.92	65.4	7.4	11.0	—	15.25	.9	.125
8	58.90	6.67	8.40	6.46	2.84	15.88	.98	62.8	10.3	7.0	3.0	15.88	1.0	.188

*Fe content is uncertain because the samples were pulverized in a steel mortar and pestle.

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APPENDIX

The following procedures were reported to Dr. L. S. Foster, Watertown Arsenal Laboratory while visiting Carboloy Company, Inc., Detroit, Michigan, Kennametal, Inc., Latrobe, Pa., and Fansteel Metallurgical Corp., Chicago, Ill., in September, 1948.

Polishing of Carbides - Carboloy Company, Inc.

1. Grind on 100 grit diamond wheel
2. Lap on a bronze metal wheel with #5 diamond dust with a light oil composed of a mixture of mineral oil or spindle oil, carbon tetrachloride. Maximum particle size of diamond powder - 10 microns.
3. Clean with soap and water
4. Polish on a paper lap glued to a metal disk with #7 diamond dust using same light oil.
   Paper lap - single weight photographic paper mounted emulsion side down. Bond paper can be used.
   Maximum particle size of diamond powder - 3 microns.
5. The final polish is accomplished on a similar paper lap using a still finer diamond powder.

Polishing of Carbides - Kennametal, Inc.

1. Grind on 220 grit resinoid bonded diamond cup wheel
2. Clean with soap and water
3. Polish on a lead-tin alloy lap using U. S. National Bureau of Standards #14 diamond dust (8-20 microns)
   Lap is a standard Eberbach for their polishing tables.
   Speed of lap - 475 rpm.
4. Clean with soap and water
5. Polish on another lead-tin alloy lap using U. S. National Bureau of Standards #1 diamond dust (0-2 microns)
   Speed of lap - 210 rpm.

Polishing of Carbides - Fansteel Metallurgical Corporation

1. Lap the specimens flat on a 100 mesh diamond impregnated wheel
2. Mount the specimens in lucite. To insure flatness in polishing each block should carry three or more pieces
3. Rough polish on a cast iron lap with 300 mesh boron carbide
4. Polish on a hardwood lap with fine diamond dust and lapping oil
5. Finish polish on a second wood lap with the finest diamond dust and kerosene.

SAMPLE NO. 1.

Fig. 1.

Unetched. X250.

Uniform distribution of
medium coarse and fine porosity.

Fig. 2.

Unetched. X1000.

Fine porosity and some
fine nonmetallic inclusions.

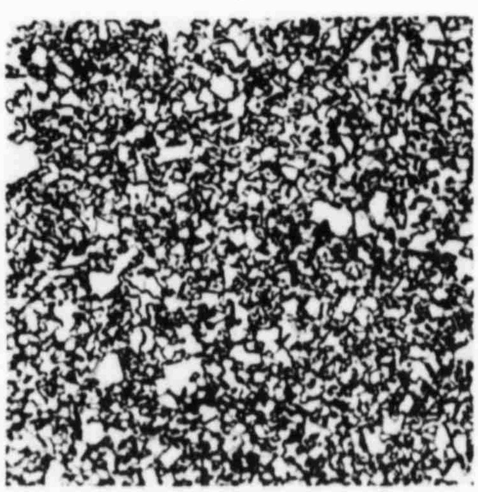

Fig. 3.

Etched. X1500.

Small and medium size angular
tungsten carbides (1), small amount
of cobalt binder (white areas).

Etchant-Hot alkaline potassium ferricyanide.

SAMPLE NO. 2.

Fig. 4.

Unetched. X250.

Uniform distribution of medium coarse and fine porosity.

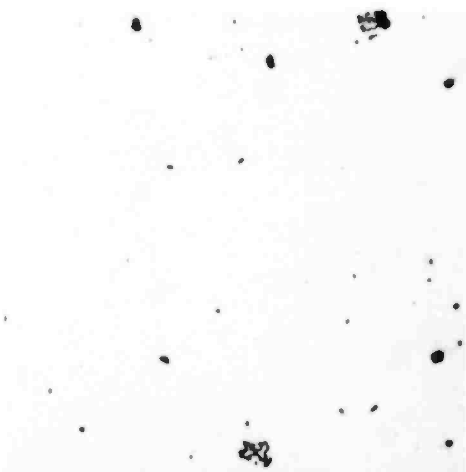

Fig. 5.

Unetched. X1000.

Few fine nonmetallic inclusions. Fine porosity and some phases in relief.

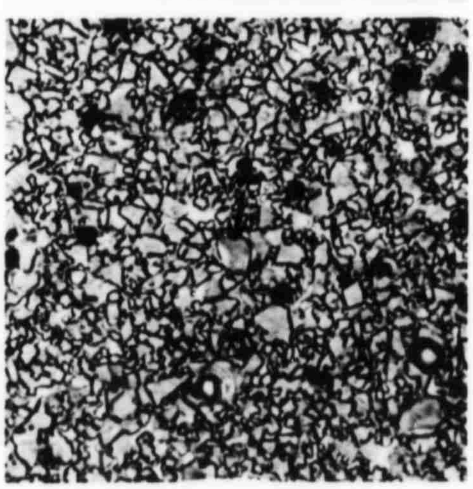

Fig. 6.

Etched. X1500.

Uniform angular tungsten carbide (1), solid solution phase (2), small amount of cobalt binder (white areas).
Note: Local attack by etching reagent.

Etchant-Hot alkaline potassium ferricyanide.

WTN.630-9717

SAMPLE NO. 3.

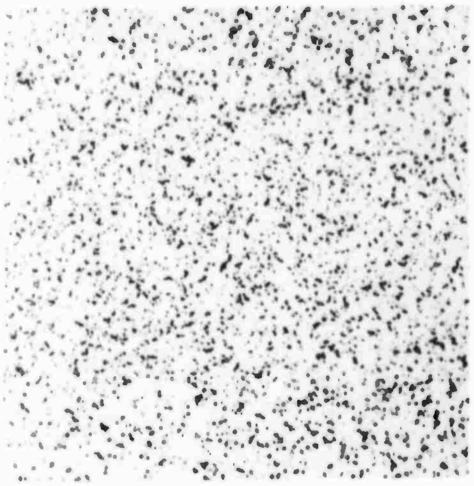

Fig. 7.

Unetched. X250.

Trace of fine porosity.
Several phases shown in relief.

Fig. 8.

Unetched. X1000.

Trace of fine porosity.
Several phases shown in relief.
Few fine nonmetallic inclusions.

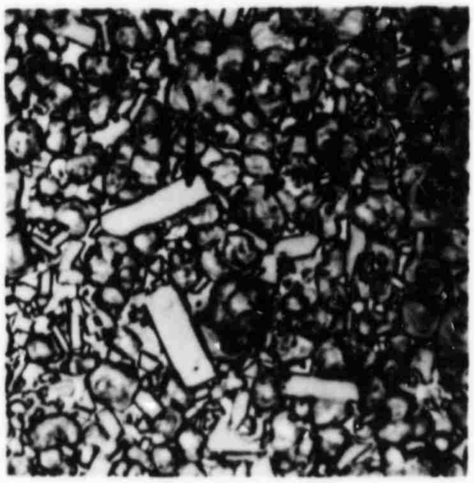

Fig. 9.

Etched. X1500.

Fairly large angular tungsten carbide (1), solid solution phase (2), small amount of cobalt binder (white areas).

Etchant-Hot alkaline potassium ferricyanide.

WTN.630-9713

SAMPLE NO. 4

Fig. 10.

Unetched. X250.

Trace of fine porosity.
Several phases shown in relief.

Fig. 11.

Unetched. X1000.

Trace of fine porosity.
Several phases shown in relief.
Few fine nonmetallic inclusions.

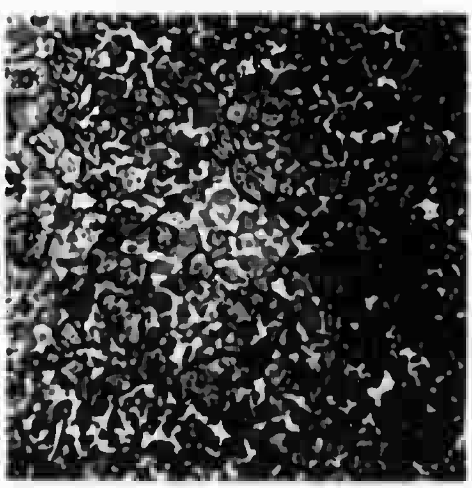

Fig. 12.

Etched. X1500.

Trace of porosity. Small angular tungsten carbide (1), solid solution phase (2), appearance of a eutectic (3), cobalt binder (white).

Etchant-Hot alkaline potassium ferricyanide.

WTN.639-0714

SAMPLE NO. 5.

Fig. 13.

Unetched. X250.

Fine porosity.

Fig. 14.

Unetched. X1000.

Few fine nonmetallic inclusions. Fine porosity and some phases shown in relief.

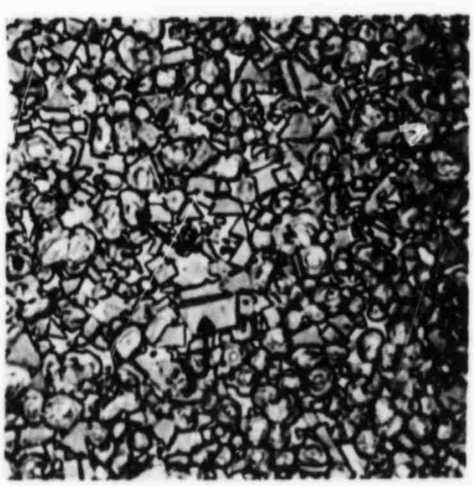

Fig. 15.

Etched. X1500.

Trace of porosity. Small and medium size angular tungsten carbide (1), solid solution phase (2), and small amount of cobalt binder (white).

Etchant-Hot alkaline potassium ferricyanice.

WTN.639-9715

SAMPLE NO. 6.

Fig. 16.

Unetched. X250.

Fine porosity and some phases shown in relief.

Fig. 17.

Unetched. X1000.

Few fine nonmetallic inclusions. Some phases shown in relief.

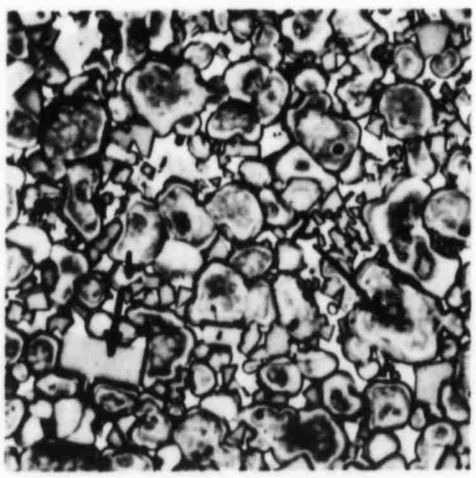

Fig. 18.

Etched. X1500.

Fairly large tungsten carbide (1), Solid solution phase (2), cobalt binder (white).

Etchant-Hot alkaline potassium ferricyanide.

WTN.639-9716

SAMPLE NO. 7.

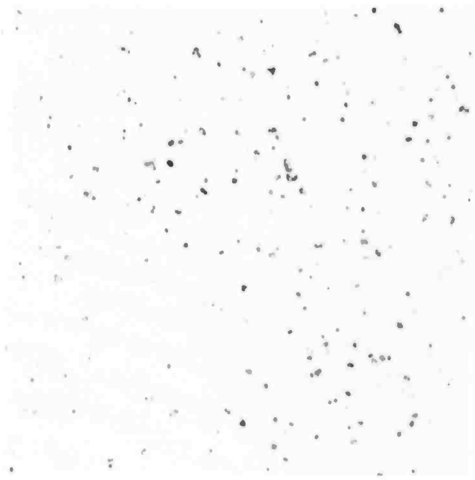

Fig. 19.

Unetched. X250.

Trace of fine porosity.
Some phases shown in relief.

Fig. 20.

Unetched. X1000.

Trace of fine porosity.
Some phases shown in relief.
Few fine nonmetallic inclusions.

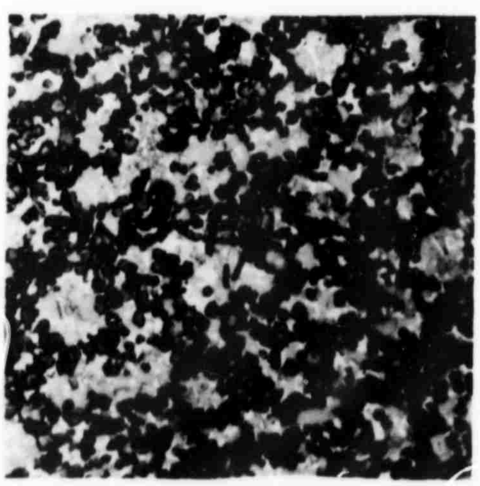

Fig. 21.

Etched. X1500.

Some porosity evident, small
angular tungsten carbide (1),
solid solution phase (2),
cobalt binder (white).

Etchant-Hot alkaline potassium ferricyanide.

WTN.639-9717

SAMPLE NO. 8.

Fig. 22.

Unetched. X250.

Trace of fine porosity.
Some phases shown in relief.

Fig. 23.

Unetched. X1000.

Trace of fine porosity.
Some phases shown in relief.
Few fine nonmetallic inclusions.

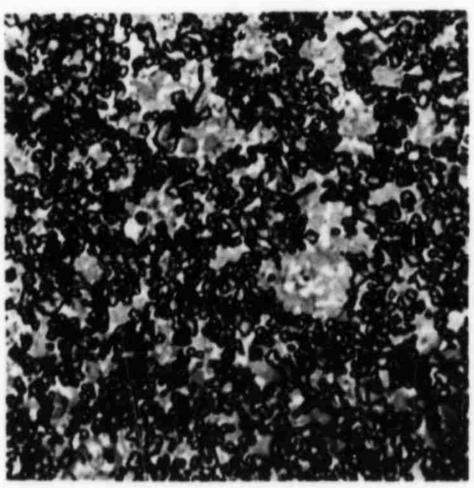

Fig. 24.

Etched. X1500.

Some porosity evident, small angular tungsten carbide (1), solid solution phase (2), cobalt binder (white).

Etchant-Hot alkaline potassium ferricyanide.

WTN.639-97/8